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# PERFORMANCE TEST OF SPECTRASPAN V SEQUENTIAL ATOMIC EMISSION SPECTROMETER

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ELLIOT LAKE LABORATORY

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# PERFORMANCE TEST OF SPECTRASPAN V SEQUENTIAL ATOMIC EMISSION SPECTROMETER

by

T.P. Lim*, N. Cloutier**, Y. Boucher** and N. Dave+

## ABSTRACT

The stability and perf[illegible]nce test of "Spectraspan V" - high energy direct current plasma emission [illegible]ntial spectrometer, was conducted.

The instrument was very stable against electronic and thermal drift components. The plasma stability of the system was very sensitive to sample introduction, electrode configuration and nebulizer pressure. At 28 psi (193 kPa) nebulizer pressure, the stability of the system lasted for over an hour compared to about 15 minutes at 24 psi (166 kPa) pressure. Regression analysis of the calibration data showed expected linearity at 95% confidence limit.

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# PERFORMANCE DU SPECTRASPAN V - SPECTROMÈTRE D'ÉMISSION ATOMIQUE À SÉQUENCES

par

T.P. Lim*, N. Cloutier**, Y. Boucher** et N. Dave+

## RÉSUMÉ

On a fait des essais visant à éprouver la stabilité et la performance du "Spectraspan V" - un spectromètre séquentiel de haute puissance à émission directe de plasma par courant continu.

Les éléments de dérive électronique et thermique n'ont pas affecté la stabilité de l'appareil. La stabilité du système en ce qui à trait au plasma était très sensible à l'introduction d'échantillons, à la configuration des électrodes et à la pression de l'atomiseur. À une pre sion de l'atomiseur de 28 livres par pouce carré (193 kPa), la stabilité du système s'est maintenue pendant plus d'une heure comparativement à environ 15 min à une pression de 24 livres par pouce carré (166 kPa). L'analyse de régression des données de calibrage à indiqué la courbe linéaire attendue à une limite de confiance de 95%.

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# INTRODUCTION

In order to improve the analytical capabilities of the surface environmental group at the Mining Research Laboratories, Elliot Lake, Ontario, an atomic emission spectrometer "Spectraspan V" with a high energy direct current plasma excitation source (DCP) has been purchased. Routinely, about 10,000 to 12,000 analyses per year are to be performed for elements such as Fe, Ca, Mg, Al, Pb, Mn, U, Th, Si, K, Ni, Co, Cu, etc., on tailings area samples dealing with the study of migration of contaminants from uranium and other tailings. It is expected that with the DCP spectrometer the total analysis time will be considerably reduced.

The "Spectraspan V" is a sequential direct current plasma emission spectrometer employing a three electrode configuration as a plasma excitation source and an echelle grating monochromator. The grating is used in combination with a quartz prism for order separation and provides a two-directional spectral pattern covering 190 nm to 800 nm. With the diffraction of collimated incident radiation the peak efficiency of all wavelengths occurs in a succession of different orders, which are vertically stacked and within these orders the wavelengths are dispersed horizontally. The echelle system has a focal length of only 0.75 m and provides an average reciprocal linear dispersion of 0.12 nm/mm and an average resolution of 0.003 nm. The detection system on a sequential spectrometer uses a single photomultiplier tube and a wavelength is selected by moving the grating and prism with a calibrated scale. The two dimensional spectrum can also be photographed using a Polaroid camera attachment to permit spectrographic qualitative analysis.

During routine operation of the DCP spectrometer, it was observed that the overall stability of the instrument was quite short-lived, sometimes as short as 10 to 15 min. Therefore, a study was undertaken to identify various

factors affecting the instrumental stability, and to test its performance for calibration characteristics, precision, accuracy, and their variations with time.

## PROCEDURE

The instrument was installed and set up according to the procedure described in the instruction manual for the Spectraspan V (1-3). The overall system stability was monitored for time variations in:

i) Electronic stability

ii) Optical and thermal stability

iii) Plasma stability.

### ELECTRONIC STABILITY

The electronic or detector stability test was performed using the built-in detector stability test light emitting diode (LED). The procedure consisted of measuring the detector output as a function of time with the LED turned on. The instrument together with the detector photomultiplier tube (PMT) high voltage and LED were first switched on. A minimum of 15 min time was allowed for warm-up and for the LED to reach stable equilibrium conditions. The mode function switch was then placed in the integrate mode with the time and repeat functions selected for 5 sec integration with 9 repetitions. The instrument was then set in the autorange analysis mode with values of 100 and 0 selected for high and low standards, respectively. After completion of the high standard cycle, the PMT high voltage was switched off and the low standard cycle was initiated to calibrate the instrument for reading the LED output light intensity as 100. The PMT voltage was switched back on and the detector output was measured at various time intervals by pressing the sample switch.

OPTICAL AND THERMAL STABILITY

For the optical and thermal stability test, a mercury lamp was used to monitor variations in the optical alignment of the instrument and its electronic stability against radiant heat that is produced by the plasma source. The procedure used was as follows:

- the mercury lamp was positioned in front of the entrance slit, between the plasma source and optical casing;
- the entrance and exit slits were adjusted to their smallest sizes;
- the lamp was turned on and the wavelength selection scroll was positioned and peaked at the 253.6 nm mercury line;
- the high and low standard values were selected as 1000 and 0, respectively, and the instrument was set in the autorange calibration mode with the function switches set for 5 sec integration and 3 repetitions;
- for the low standard, the entrance slit was blocked off with an opaque plate;
- with the mercury lamp on, the detector output was monitored every minute for about 40 min without the plasma on;
- after the mercury lamp had reached equilibrium conditions, which was approximately 40 min, the plasma torch was turned on and its light blocked off with an opaque plate, such that only the light from the mercury lamp reached the entrance slit;
- with the plasma source on, the detector output was again monitored with time;
- if a decrease in the detector output was observed, at a certain time the optical alighment was re-adjusted and the drift measured.
- for this test the fluctuations in the room temperature were also measured simultaneously.

PLASMA STABILITY

The plasma stability of the instrument was measured from its overall stability which was monitored in a normal running mode by aspirating a standard solution and measuring the observed concentration as a function of time. The instrument was first calibrated and then concentrations of various standard solutions were measured at one minute intervals. The routine procedure followed is summarized as follows:

- The plasma source was first cleaned including the nebulizer, glass sample tube, etc., the electrodes and sleeves replaced and the various components realigned. This general maintenance procedure was followed every morning in general.
- The plasma was then established and the equipment was left to run idle with distilled water aspirated for 15 min to reach thermal equilibrium conditions. The plasma was then positioned approximately at the entrance of the input slit.
- The required entrance and exit slit sizes were selected before peaking the instrument.
- A peaking solution containing the desired element was aspirated into the plasma. Depending upon the concentration range of the samples to be analyzed, a suitable spectral line for that element was selected and its position optimized by adjusting the horizontal and vertical scroll thumbwheels for maximum detector output signal (1-4). The plasma position was further aligned by adjusting the x-y controls on the jet assembly to maximize the peak. The peaking operation was repeated whenever the slit sizes were changed.
- Using the numeric key pad, the calibration values for high and low standards were entered. The high standard was then aspirated into the plasma and the instrument was set in the auto range mode with 5 sec

integration and 3 repetitions. The auto range cycle automatically adjusted the instrument gain near mid-range. Upon completion of the auto range cycle, the low standard solution was aspirated, and a minimum of 15 sec time was allowed for rinsing of the sample delivery system before pressing the low standard calibration button.

- After the instrument was calibrated, several standard solutions with concentrations ranging between the two calibration standards were aspirated for a period of one hour to monitor instrumental precision, accuracy and stability.
- The above tests were repeated for various elements such as calcium, iron and aluminium, etc., commonly found in groundwater samples.

## RESULTS AND DISCUSSION

Table 1 shows the results for the electronic and detector stability test. For an assigned test current value of 100, the measured value was 99.4 $\pm$ 0.5. For a period of one hour, the overall fluctuations in the mean value were $\pm$0.5%, signifying a good electronic stability of the system.

The optical and thermal stability test results are shown in Fig. 1. The results showed that approximately 40 min were required for the mercury lamp to reach thermal equilibrium. After the plasma torch was turned on, the detector output started to decrease by approximately 8% in the first 20 min as the instrument shifted to new thermal equilibrium conditions with the dissipation of the plasma heat. The system reached thermal equilibrium in one hour after the plasma had been started. Beyond that, a slight drift of approximately 10% in one hour was observed. It was also observed during the test that the wavelength selection (optical alignment) had shifted which required only vertical re-adjustment of the scroll for repeating the detector output.

The ambient temperature of the DCP room was monitored for several days and during a typical operating period the room temperature fluctuations did not exceed $\pm 1^{o}C/h$ as shown in Fig. 2.

The overall stability of the instrument in terms of variations in the measured concentration of a standard solution are shown in Table 2 and Fig. 3 for Ca, and Tables 3,4 and Fig. 4 and 5 for Fe at 24 psi nebulizer pressure. It is seen from these results that the overall stability at 24 psi was very shortlived, lasting only between 10-15 min after peaking and auto range calibration. Thereafter the stability dropped at the rate of approximately 5-10% in 15 min. This instability was primarily caused by plasma related fluctuations as the electronic and thermal stability of the system were good.

Tables 5, 6 and 7, and Fig. 6 and 7 show the observed stabilities for Ca, Al and Fe at 28 psi nebulizer pressure. The results clearly show a much improved stability lasting over a period of one hour, compared to that observed at 24 psi nebulizer pressure. During these tests, it was observed that sample introduction, nebulizer pressure, electrode gap, condition and alignment were very important factors contributing to the plasma related stability of the system. Nebulizer pressure was critical in stable plasma formation, sample introduction and aerosol formation. Increasing nebulizer pressure beyond 28 psi produced improper sample aerosols consisting of inhomogeneous large sample droplets which gave erratic results. Sample introduction is very important in plasma emission spectrometry and is discussed in detail by Browner and Boorn (5).

The calibration data for Ca, Fe and Al are given in Tables 8 to 10 and are plotted in Fig. 8 to 10, respectively. Tables 8 to 10 also give linear regression of the observed data, calculated concentrations and t-test on the observed slope and intercept (6). The results showed no significant difference between observed and expected values at 95% confidence limit.

The overall performance of the instrument was satisfactory once a daily operating maintenance routine, consisting of changing and aligning electrodes; cleaning of sample introduction tubes and nebulizer, had been carried out.

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4. Spectrametric Incorporated, "Handbook of spectral line characteristics for the DC plasma/echelle systems"; Andover, Mass, 1983.

5 Browner, R.F. and Boorn, A.W., "Sample introduction"; Analytical Chemistry, vol 50, No. 7, pp 786-798, June 1984; and "Sample introduction techniques for atomic spectroscopy"; Ibid., pp 875A-888A.

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## Table 1 - Electronic and detector stability test using built-in light emitting diode

```
TIME=10 REP= 9 SAMPLE=  19
 1  100. 99.9 100. 100. 99.7 99.2 100. 100. 99.5 AV 100. SD .424

 1 0048 0143 4865 0000 0100 4883 4863 4883 4877 4851 4831 4888 4867 4842


TIME=10 REP= 9 SAMPLE=  20
 1  99.8 100. 100. 99.2 99.9 100. 100. 100. 99.9 AV 100. SD .362

 1 0048 0143 4865 0000 0100 4859 4869 4888 4829 4861 4870 4879 4880 4861

TIME=10 REP= 9 SAMPLE=  21
 1  100. 100. 99.9 100. 99.5 99.6 100. 99.1 99.2 AV 99.9 SD .641

 1 0048 0143 4865 0000 0100 4907 4905 4863 4867 4842 4846 4880 4825 4829

TIME=10 REP= 9 SAMPLE=  22
 1  100. 100. 99.5 100. 99.9 99.4 99.2 100. 100. AV 99.9 SD .440

 1 0048 0143 4865 0000 0100 4879 4868 4842 4879 4863 4840 4831 4884 4885

TIME=10 REP= 9 SAMPLE=  23
 1  99.5 99.3 99.3 99.4 99.9 100. 99.7 99.9 98.9 AV 99.6 SD .406

 1 0048 0143 4865 0000 0100 4843 4835 4833 4841 4864 4877 4854 4862 4814

TIME=10 REP= 9 SAMPLE=  24
 1  100. 99.8 100. 99.6 100. 99.8 99.8 98.5 98.7 AV 99.7 SD .661

 1 0048 0143 4865 0000 0100 4870 4857 4883 4850 4883 4860 4857 4796 4804

TIME=10 REP= 9 SAMPLE=  25
 1  98.5 99.1 99.8 99.1 99.3 99.8 99.3 100. 99.8 AV 99.4 SD .506

 1 0048 0143 4865 0000 0100 4796 4824 4856 4823 4835 4860 4835 4872 4859

TIME=10 REP= 9 SAMPLE=  26
 1  99.7 99.6 99.6 99.0 98.4 99.0 99.1 99.2 99.1 AV 99.2 SD .398

 1 0048 0143 4865 0000 0100 4851 4846 4850 4820 4791 4822 4825 4828 4823

TIME=10 REP= 9 SAMPLE=  27
 1  99.2 99.0 98.5 99.3 100. 99.1 99.7 99.8 99.1 AV 99.3 SD .549

 1 0048 0143 4865 0000 0100 4831 4819 4795 4835 4885 4824 4854 4857 4827

TIME=10 REP= 9 SAMPLE=  28
 1  99.3 99.6 98.9 99.2 99.4 99.7 99.4 99.2 99.8 AV 99.4 SD .288

 1 0048 0143 4865 0000 0100 4835 4850 4815 4828 4841 4851 4838 4829 4859

TIME=10 REP= 9 SAMPLE=  29
 1  99.6 99.0 99.3 98.8 98.6 99.1 99.1 98.0 99.1 AV 98.9 SD .441

 1 0048 0143 4865 0000 0100 4848 4818 4832 4810 4802 4823 4824 4774 4826

TIME=10 REP= 9 SAMPLE=  30
 1  99.2 99.0 99.7 99.0 99.3 98.9 98.9 99.4 99.2 AV 99.2 SD .272

 1 0048 0143 4865 0000 0100 4828 4818 4854 4820 4834 4813 4816 4837 4828
```

| Raw Data | Assigned Value |
|---|---|
| Mean = 4847.4 | 99.4 |
| Standard deviation ±26.7 | ±0.5 |
| Standard error ±0.5% | ±0.5% |

Table 2 - Stability test on calcium line at 317.9 nm observed at 100 ppm concentration and nebulizer pressure at 24 psi.

| Time (min) | Standard (ppm) | Calcium 1 | Calcium 2 | Calcium 3 | Calcium Ave. | Raw Data 1 | Raw Data 2 | Raw Data 3 |
|---|---|---|---|---|---|---|---|---|
| 0 | 100 | - | - | - | - | 4677 | 4554 | 4691 |
| 0 | 0 | - | - | - | - | 125 | 123 | 124 |
| 3 | 100 | 103.0 | 108.0 | 103.0 | 105.0 | 4804 | 5001 | 4794 |
| 4 | 100 | 109.0 | 105.0 | 109.0 | 108.0 | 5082 | 4892 | 5083 |
| 5 | 100 | 108.0 | 108.0 | 107.0 | 108.0 | 5015 | 5009 | 4977 |
| 6 | 100 | 104.0 | 102.0 | 103.0 | 103.0 | 4853 | 4736 | 4818 |
| 7 | 100 | 104.0 | 105.0 | 100.0 | 103.0 | 4832 | 4880 | 4674 |
| 8 | 100 | 105.0 | 104.0 | 102.0 | 104.0 | 4881 | 4832 | 4755 |
| 9 | 100 | 106.0 | 107.0 | 107.0 | 106.0 | 4911 | 4956 | 4979 |
| 10 | 100 | 107.0 | 107.0 | 109.0 | 107.0 | 4965 | 4968 | 5067 |
| 11 | 100 | 102.0 | 105.0 | 104.0 | 104.0 | 4741 | 4892 | 4860 |
| 12 | 100 | 104.0 | 107.0 | 105.0 | 105.0 | 4825 | 4983 | 4896 |
| 13 | 100 | 100.0 | 104.0 | 105.0 | 103.0 | 4651 | 4846 | 4909 |
| 14 | 100 | 104.0 | 106.0 | 104.0 | 105.0 | 4840 | 4923 | 4860 |
| 15 | 100 | 106.0 | 106.0 | 105.0 | 106.0 | 4854 | 4937 | 4967 |
| 16 | 0 | 0.642 | 0.044 | 0.022 | 0.236 | 153 | 126 | 125 |
| 17 | 0 | 0.0 | 0.0 | 0.022 | 0.007 | 124 | 124 | 125 |
| | | Maximized + Repeaked + Auto ranged | | | | | | |
| 20 | 100 | - | - | - | - | 4971 | 4890 | 4835 |
| 20 | 100 | - | - | - | - | 133 | 123 | 122 |
| 23 | 100 | 102.0 | 103.0 | 102.0 | 102.0 | 5014 | 5071 | 5019 |
| 24 | 100 | 101.0 | 100.0 | 98.2 | 99.8 | 4948 | 4904 | 4817 |
| 25 | 100 | 98.4 | 99.5 | 99.6 | 99.2 | 4823 | 4878 | 4884 |
| 26 | 100 | 99.7 | 101.0 | 100.0 | 100.0 | 4888 | 4857 | 4926 |
| 27 | 100 | 101.0 | 99.5 | 99.6 | 100.0 | 4983 | 4877 | 4881 |
| 28 | 100 | 100.0 | 99.5 | 100.0 | 100.0 | 4942 | 4875 | 4937 |
| 29 | 100 | 100.0 | 99.0 | 100.0 | 99.7 | 4908 | 4854 | 4902 |
| 36 | 100 | 100.0 | 100.0 | 99.8 | 100.0 | 4921 | 4924 | 4893 |
| 31 | 100 | 99.8 | 96.5 | 97.6 | 98.0 | 4894 | 4732 | 4785 |
| 32 | 0 | 0.083 | -0.041 | -0.041 | 0.0 | 130 | 124 | 124 |
| 33 | 0 | -0.041 | -0.041 | -0.062 | -0.048 | 124 | 124 | 123 |
| 34 | 0 | -0.041 | -0.041 | -0.034 | -0.034 | 124 | 124 | 125 |
| 35 | 0 | -0.062 | -0.062 | -0.041 | -0.055 | 123 | 123 | 124 |
| 36 | 100 | 94.8 | 94.7 | 95.8 | 95.1 | 4654 | 4650 | 4701 |
| 37 | 100 | 96.3 | 96.4 | 96.4 | 96.4 | 4723 | 4730 | 4731 |
| 38 | 100 | 93.5 | 95.6 | 94.3 | 94.5 | 4590 | 4692 | 4631 |
| 39 | 100 | 94.9 | 95.8 | 94.8 | 95.2 | 4656 | 4700 | 4655 |
| 40 | 0 | 0.021 | -0.021 | -0.021 | -0.007 | 127 | 125 | 125 |
| 41 | 0 | -0.041 | -0.041 | -0.021 | -0.034 | 124 | 124 | 125 |
| 42 | 0 | -0.041 | -0.021 | -0.041 | -0.034 | 124 | 125 | 124 |
| 43 | 100 | 97.5 | 96.5 | 96.9 | 97.0 | 4780 | 4735 | 4752 |
| 44 | 100 | 95.6 | 95.5 | 96.5 | 95.9 | 4693 | 4685 | 4734 |
| 45 | 100 | 95.9 | 96.0 | 96.6 | 96.2 | 4704 | 4708 | 4741 |
| 47 | 100 | 94.4 | 90.9 | 90.3 | 91.9 | 4635 | 4466 | 4440 |
| 48 | 100 | 89.7 | 90.3 | 94.6 | 91.5 | 4411 | 4439 | 4643 |
| 49 | 100 | 96.7 | 97.3 | 98.3 | 97.4 | 4742 | 4770 | 4818 |

Cont. overleaf

Table 2 - Cont.

| Time (min) | Standard (ppm) | Calcium 1 | 2 | 3 | Ave. | Raw Data 1 | 2 | 3 |
|---|---|---|---|---|---|---|---|---|
| 50 | 100 | 95.5 | 94.2 | 95.0 | 94.9 | 4686 | 4623 | 4661 |
| 51 | 100 | 93.4 | 95.2 | 98.5 | 95.7 | 4584 | 4671 | 4828 |
| 52 | 100 | 93.0 | 92.6 | 95.7 | 93.8 | 4566 | 4550 | 4695 |
| | | Maximized + Repeak + Auto Ranged | | | | | | |
| 55 | 100 | - | - | - | - | 4516 | 4621 | 4701 |
| 55 | 0 | - | - | - | - | 133 | 124 | 125 |
| 58 | 100 | 103.0 | 103.0 | 103.0 | 103.0 | 4765 | 4763 | 4773 |
| 59 | 100 | 101.0 | 103.0 | 103.0 | 103.0 | 4668 | 4783 | 4788 |
| 60 | 100 | 101.0 | 103.0 | 102.0 | 102.0 | 4683 | 4756 | 4720 |
| 61 | 100 | 99.5 | 100.0 | 102.0 | 100.0 | 4594 | 4618 | 4720 |
| 62 | 100 | 100.0 | 100.0 | 100.0 | 100.0 | 4646 | 4635 | 4646 |
| 63 | 100 | 103.0 | 101.0 | 100.0 | 101.0 | 4774 | 4677 | 4641 |
| 64 | 100 | 106.0 | 102.0 | 105.0 | 104.0 | 4901 | 4739 | 4847 |
| 65 | 100 | 104.0 | 101.0 | 101.0 | 102.0 | 4791 | 4685 | 4692 |
| 66 | 0 | 0.022 | -0.044 | -0.066 | -0.029 | 128 | 125 | 124 |
| 67 | 0 | -0.044 | -0.044 | -0.066 | -0.055 | 125 | 125 | 124 |
| 68 | 100 | 103.0 | 103.0 | 102.0 | 103.0 | 4766 | 4790 | 4731 |
| 69 | 100 | 104.0 | 103.0 | 101.0 | 102.0 | 4807 | 4761 | 4660 |
| 70 | 100 | 104.0 | 103.0 | 103.0 | 103.0 | 4822 | 4760 | 4760 |
| 71 | 100 | 100.0 | 104.0 | 103.0 | 102.0 | 4637 | 4818 | 4768 |
| 72 | 100 | 106.0 | 107.0 | 105.0 | 106.0 | 4882 | 4925 | 4869 |
| 73 | 0 | 0.0 | -0.066 | -0.089 | -0.052 | 127 | 124 | 123 |
| 75 | 0 | -0.066 | -0.066 | -0.066 | -0.066 | 124 | 124 | 124 |
| 77 | 100 | 101.0 | 101.0 | 103.0 | 102.0 | 4670 | 4788 | 4712 |
| 78 | 100 | 103.0 | 101.0 | 103.0 | 103.0 | 4776 | 4688 | 4781 |
| 79 | 100 | 101.0 | 98.7 | 102.0 | 101.0 | 4687 | 4555 | 4730 |
| 80 | 100 | 100.0 | 99.9 | 102.0 | 101.0 | 4623 | 4611 | 4735 |
| 81 | 0 | -0.022 | -0.044 | -0.089 | -0.052 | 126 | 125 | 123 |
| 82 | 0 | -0.066 | -0.111 | -0.111 | -0.096 | 124 | 122 | 122 |
| 83 | 0 | -0.066 | -0.089 | -0.066 | -0.074 | 124 | 123 | 124 |
| 84 | 100 | 102.0 | 101.0 | 103.0 | 102.0 | 4710 | 4663 | 4761 |
| 85 | 100 | 104.0 | 104.0 | 104.0 | 104.0 | 4834 | 4814 | 4791 |
| 86 | 100 | 103.0 | 105.0 | 108.0 | 105.0 | 4753 | 4872 | 4982 |
| 87 | 100 | 101.0 | 101.0 | 102.0 | 102.0 | 4693 | 4672 | 4744 |

Table 3 - Stability test on iron line at 259.9 nm observed at 100 ppm and 0 ppm concentration.

| Time (min) | Standard (ppm) | Iron | | | | Raw Data | | |
|---|---|---|---|---|---|---|---|---|
| | | 1 | 2 | 3 | Ave. | 1 | 2 | 3 |
| 0 | 100 | - | - | - | - | 4675 | 4743 | 4703 |
| 0 | 0 | - | - | - | - | 132 | 119 | 120 |
| 3 | 100 | 95.3 | 99.3 | 98.1 | 97.6 | 4495 | 4679 | 4624 |
| 4 | 100 | 100.0 | 106.0 | 105.0 | 104.0 | 4747 | 5015 | 4977 |
| 5 | 100 | 105.0 | 101.0 | 100.0 | 102.0 | 4954 | 4790 | 4736 |
| 6 | 100 | 101.0 | 97.9 | 105.0 | 101.0 | 4773 | 4615 | 4972 |
| 7 | 0 | -0.109 | -0.109 | -0.087 | -0.080 | 121 | 118 | 119 |
| 8 | 0 | -0.065 | -0.087 | -0.109 | -0.087 | 120 | 119 | 118 |
| 9 | 0 | -0.087 | -0.109 | -0.087 | -0.094 | 119 | 118 | 119 |
| 10 | 100 | 102.0 | 99.3 | 100.0 | 100.0 | 4800 | 4679 | 4715 |
| 11 | 100 | 99.1 | 106.0 | 103.0 | 102.0 | 4669 | 4992 | 4864 |
| 12 | 100 | 102.0 | 107.0 | 95.5 | 101.0 | 4836 | 5026 | 4503 |
| 13 | 100 | 98.8 | 95.2 | 98.4 | 97.4 | 4652 | 4487 | 4635 |
| 14 | 0 | -0.043 | -0.065 | -0.109 | -0.072 | 121 | 120 | 118 |
| 18 | 100 | 98.8 | 96.9 | 95.5 | 97.1 | 4656 | 4565 | 4505 |
| 19 | 100 | 92.1 | 87.4 | 91.3 | 90.3 | 4349 | 4132 | 4312 |
| 20 | 100 | 97.9 | 95.9 | 100.0 | 98.2 | 4615 | 4521 | 4738 |
| 21 | 0 | 0.087 | -0.021 | -0.065 | -0.000 | 127 | 122 | 120 |
| 22 | 0 | -0.065 | -0.065 | -0.065 | -0.065 | 120 | 120 | 120 |
| 23 | 0 | -0.021 | -0.021 | -0.021 | -0.021 | 122 | 122 | 122 |
| 24 | 100 | 85.0 | 89.5 | 89.3 | 87.9 | 4020 | 4228 | 4218 |
| 25 | 100 | 88.3 | 84.4 | 85.2 | 85.9 | 4171 | 3994 | 4029 |
| 26 | 100 | 87.8 | 86.1 | 86.0 | 86.6 | 4151 | 4072 | 4068 |
| 27 | 0 | -0.065 | -0.087 | -0.087 | -0.080 | 120 | 119 | 119 |
| 28 | 0 | -0.087 | -0.087 | -0.109 | -0.094 | 119 | 119 | 118 |
| 29 | 100 | 75.6 | 78.3 | 76.7 | 76.8 | 3590 | 3713 | 3639 |
| 30 | 100 | 79.4 | 77.8 | 79.8 | 79.0 | 3767 | 3693 | 3785 |
| 31 | 0 | -0.043 | -0.065 | -0.087 | -0.065 | 121 | 120 | 119 |
| 32 | 0 | -0.087 | -0.087 | -0.109 | -0.094 | 119 | 119 | 118 |

Table 4 - Stability test on iron line at 259.9 nm for various concentrations between 0-1000 ppm.

| Actual Conc. Fe(ppm) | Observed Conc. Fe(ppm) | Deviation from Actual | Observed Raw Data | Calc. Conc. Based on $Y=aX^b+C$ | Deviation of Calc. from Actual Conc. | Time (min) after Calib. |
|---|---|---|---|---|---|---|
| 250 | 242.0 | -8.0 | 1280 | 233.3 | -16.8 | 10 |
| 800 | 756.0 | -44.0 | 3760 | 782.9 | -17.1 | 12 |
| 10 | 11.3 | +1.3 | 164 | 9.0 | -1.0 | 14 |
| 500 | 503.0 | +3.0 | 2542 | 508.2 | +8.2 | 16 |
| 100 | 108.0 | +8.0 | 632 | 98.9 | -1.1 | 18 |
| 0 | 0.069 | +0.069 | 109.3 | 0.0391 | +0.0391 | 20 |
| 50 | 51.9 | +1.9 | 360 | 45.2 | -4.8 | 22 |
| 0 | 0.138 | +0.138 | 110 | 0.126 | +0.126 | 24 |
| 0 | 0.138 | +0.138 | 110 | 0.126 | +0.126 | 65 |
| 1000 | 821.0 | -179.0 | 4076 | 855.2 | -144.8 | 66 |
| 250 | 200.0 | -50.0 | 1077 | 190.5 | -59.5 | 68 |
| 10 | 8.28 | -1.72 | 149 | 6.4 | -3.6 | 70 |
| 1000 | 788.0 | -212.0 | 3917 | 818.8 | -181.2 | 72 |
| 500 | 428.0 | -72.0 | 2176 | 427.2 | -72.8 | 74 |
| 800 | 643.0 | -157.0 | 3215 | 659.0 | -141.0 | 76 |
| 50 | 43.4 | -6.6 | 319 | 37.4 | -12.6 | 78 |
| 100 | 89.8 | -10.2 | 543 | 81.1 | -18.9 | 80 |
| 100 | 95.0 | -5.0 | 568 | 86.0 | -14.0 | 82 |
| 0 | -0.069 | -0.069 | 109 | -0.0350 | -0.0350 | 84 |
| 500 | 402.0 | -98.0 | 2051 | 399.7 | -100.3 | 86 |
| 100 | 81.4 | -18.6 | 502 | 72.9 | -27.1 | 88 |
| 800 | 581.0 | -219.0 | 2914 | 591.3 | -208.7 | 90 |
| 10 | 8.69 | -1.31 | 151 | 6.7 | -3.3 | 92 |
| 100 | 82.3 | -17.7 | 506 | 73.7 | -26.3 | 94 |
| 250 | 199.0 | -51.0 | 1071 | 189.2 | -60.8 | 96 |
| 1000 | 670.0 | -330.0 | 3347 | 688.9 | -311.1 | 98 |
| 50 | 38.8 | -11.2 | 296 | 33.1 | -16.9 | 100 |
| 500 | 350.0 | -150.0 | 1802 | 345.4 | -154.6 | 108 |
| 10 | 7.11 | -2.89 | 143 | 5.4 | -4.6 | 110 |
| 1000 | 642.0 | -358.0 | 3210 | 657.9 | -342.1 | 112 |
| 800 | 514.0 | -286.0 | 2594 | 519.7 | -280.3 | 114 |
| 50 | 31.6 | -18.4 | 262 | 26.7 | -23.3 | 116 |
| 250 | 160.0 | -90.0 | 882 | 149.9 | -100.1 | 118 |
| 0 | 0 | 0 | 109 | 0 | 0 | 120 |
| 100 | 71.1 | -28.9 | 452 | 63.1 | -36.9 | 122 |

Table 5 - Calcium stability test at 317.9 nm using 100 ppm standard solution and 28 psi nebulizing pressure

| Time (min) | Standard Calcium Assigned 100 | Observed Calcium Reading | Calculated Calcium Reading | Standard Deviation from the Mean |
|---|---|---|---|---|
| II | | Y(II) | YC(II) | YS(II) |
| 1 | | 101.00000 | 102.20870 | 0.61596 |
| 2 | | 102.22000 | 103.14545 | 0.67555 |
| 3 | | 101.00000 | 100.21898 | 0.58492 |
| 4 | | 100.22222 | 101.03245 | 0.56458 |
| 5 | | 102.02000 | 101.79621 | 0.54456 |
| 6 | | 102.00222 | 101.68852 | 0.52488 |
| 7 | | 101.22222 | 101.51305 | 0.52560 |
| 8 | | 100.22222 | 101.38056 | 0.48875 |
| 9 | | 101.00222 | 101.28028 | 0.46842 |
| 10 | | 100.22202 | 101.13350 | 0.45002 |
| 11 | | 101.00222 | 101.02710 | 0.43345 |
| 12 | | 100.22222 | 100.88264 | 0.41698 |
| 13 | | 101.22202 | 100.75415 | 0.40189 |
| 14 | | 102.22220 | 100.60767 | 0.38548 |
| 15 | | 101.00222 | 100.50119 | 0.37265 |
| 16 | | 99.00220 | 100.37471 | 0.35992 |
| 17 | | 100.22222 | 100.24822 | 0.34842 |
| 18 | | 101.22222 | 100.12174 | 0.33085 |
| 19 | | 99.00222 | 99.99526 | 0.32955 |
| 20 | | 99.99222 | 99.88878 | 0.31844 |
| 21 | | 100.22222 | 99.74229 | 0.31728 |
| 22 | | 99.88222 | 99.61581 | 0.31338 |
| 23 | | 101.22222 | 99.48933 | 0.31159 |
| 24 | | 99.88200 | 99.36285 | 0.31167 |
| 25 | | 100.00220 | 99.23636 | 0.31362 |
| 26 | | 99.78022 | 99.12988 | 0.31742 |
| 27 | | 100.00002 | 98.96342 | 0.32298 |
| 28 | | 99.55222 | 98.89693 | 0.33083 |
| 29 | | 98.00222 | 98.73044 | 0.33926 |
| 30 | | 97.42200 | 98.60395 | 0.34324 |
| 31 | | 98.66200 | 98.47747 | 0.36296 |
| 32 | | 99.12202 | 98.35099 | 0.37379 |
| 33 | | 97.56222 | 98.22451 | 0.38770 |
| 34 | | 98.88222 | 98.09802 | 0.40259 |
| 35 | | 98.88202 | 98.22451 | 0.38772 |
| 36 | | 98.42002 | 98.09802 | 0.40259 |
| 37 | | 96.56202 | 97.97154 | 0.41635 |
| 38 | | 97.78022 | 97.84506 | 0.43488 |
| 39 | | 97.22000 | 97.71858 | 0.45211 |
| 40 | | 97.88220 | 97.59209 | 0.46936 |
| 41 | | 95.52220 | 97.46561 | 0.48835 |
| 42 | | 97.12200 | 97.33913 | 0.50723 |
| 43 | | 98.12200 | 97.21265 | 0.52654 |
| 44 | | 95.50200 | 97.08616 | 0.54665 |
| 45 | | 97.32220 | 96.95968 | 0.56630 |
| 46 | | 98.10202 | 96.83320 | 0.58557 |
| 47 | | 95.22200 | 96.70672 | 0.60732 |

Table 6 - Aluminum stability test at 308.2 nm and 28 psi nebulizer pressure (100 ppm standard)

| Time (min) | Standard Aluminum Assigned (ppm) | Observed Aluminum (ppm) |
|---|---|---|
| II | X(II) | Y(II) |
| 1 | 0.00000 | 0.01200 |
| 2 | 100.00000 | 100.00000 |
| 3 | 100.00000 | 100.00000 |
| 4 | 100.00000 | 101.00000 |
| 5 | 100.00000 | 99.50000 |
| 6 | 100.00000 | 100.00000 |
| 7 | 100.00000 | 100.00000 |
| 8 | 100.00000 | 100.00000 |
| 9 | 50.00000 | 49.30000 |
| 10 | 50.00000 | 48.30000 |
| 11 | 50.00000 | 48.60000 |
| 12 | 0.00000 | -0.05400 |
| 13 | 100.00000 | 102.00000 |
| 14 | 100.00000 | 100.00000 |
| 15 | 100.00000 | 99.80000 |
| 16 | 100.00000 | 99.00000 |
| 17 | 100.00000 | 99.90000 |
| 18 | 100.00000 | 100.00000 |
| 19 | 100.00000 | 99.90000 |
| 20 | 100.00000 | 99.80000 |
| 21 | 50.00000 | 48.70000 |
| 22 | 50.00000 | 48.60000 |
| 23 | 50.00000 | 48.10000 |
| 24 | 50.00000 | 48.50000 |
| 25 | 50.00000 | 48.70000 |
| 26 | 100.00000 | 99.40000 |
| 27 | 100.00000 | 99.20000 |
| 28 | 100.00000 | 98.70000 |
| 29 | 100.00000 | 98.60000 |
| 30 | 100.00000 | 98.90000 |
| 31 | 100.00000 | 98.10000 |
| 32 | 100.00000 | 99.50000 |
| 33 | 100.00000 | 99.50000 |
| 34 | 100.00000 | 98.80000 |
| 35 | 100.00000 | 98.30000 |
| 36 | 100.00000 | 98.70000 |
| 37 | 100.00000 | 99.40000 |
| 38 | 100.00000 | 98.20000 |
| 39 | 100.00000 | 99.00000 |
| 40 | 100.00000 | 98.00000 |
| 41 | 100.00000 | 98.00000 |
| 42 | 100.00000 | 99.60000 |
| 43 | 100.00000 | 98.30000 |
| 44 | 100.00000 | 97.00000 |

Table 7 - Iron Stability test at 259.9 nm and 28 psi nebulizer pressure.

| Time (min) | Iron Standard Assigned (ppm) | Iron Observed (ppm) |
|---|---|---|
| II | X(II) | Y(II) |
| 1 | 0.00000 | -0.09300 |
| 2 | 0.00000 | -0.09300 |
| 3 | 0.00000 | -0.09300 |
| 4 | 900.00000 | 900.00000 |
| 5 | 900.00000 | 872.00000 |
| 6 | 900.00000 | 838.00000 |
| 7 | 900.00000 | 885.00000 |
| 8 | 900.00000 | 864.00000 |
| 9 | 900.00000 | 893.00000 |
| 10 | 900.00000 | 876.00000 |
| 11 | 900.00000 | 860.00000 |
| 12 | 900.00000 | 878.00000 |
| 13 | 900.00000 | 899.00000 |
| 14 | 900.00000 | 902.00000 |
| 15 | 900.00000 | 895.00000 |
| 16 | 900.00000 | 897.00000 |
| 17 | 900.00000 | 888.00000 |
| 18 | 900.00000 | 890.00000 |
| 19 | 900.00000 | 886.00000 |
| 20 | 900.00000 | 902.00000 |
| 21 | 900.00000 | 881.00000 |
| 22 | 450.00000 | 459.00000 |
| 23 | 450.00000 | 469.00000 |
| 24 | 450.00000 | 460.00000 |
| 25 | 450.00000 | 455.00000 |
| 26 | 450.00000 | 463.00000 |
| 27 | 450.00000 | 450.00000 |
| 28 | 200.00000 | 203.00000 |
| 29 | 200.00000 | 203.00000 |
| 30 | 200.00000 | 198.00000 |
| 31 | 100.00000 | 102.00000 |
| 32 | 100.00000 | 103.00000 |
| 33 | 100.00000 | 101.00000 |
| 34 | 50.00000 | 53.70000 |
| 35 | 50.00000 | 52.80000 |
| 36 | 50.00000 | 54.90000 |
| 37 | 50.00000 | 52.90000 |
| 38 | 50.00000 | 52.70000 |
| 39 | 50.00000 | 54.20000 |
| 40 | 25.00000 | 25.50000 |
| 41 | 25.00000 | 25.70000 |
| 42 | 25.00000 | 26.20000 |
| 43 | 10.00000 | 10.80000 |
| 44 | 10.00000 | 11.20000 |
| 45 | 10.00000 | 11.20000 |

Cont. overleaf

Table 7 - Cont.

| Time (min) | Iron Standard Assigned (ppm) | Iron Observed (ppm) |
|---|---|---|
| 46 | 5.00000 | 5.21000 |
| 47 | 5.00000 | 5.11000 |
| 48 | 5.00000 | 5.39000 |
| 49 | 450.00000 | 439.00000 |
| 50 | 450.00000 | 454.00000 |
| 51 | 450.00000 | 453.00000 |
| 52 | 900.00000 | 686.00000 |
| 53 | 900.00000 | 865.00000 |
| 54 | 900.00000 | 874.00000 |
| 55 | 900.00000 | 890.00000 |
| 56 | 900.00000 | 869.00000 |
| 57 | 900.00000 | 882.00000 |
| 58 | 5.00000 | 5.77000 |
| 59 | 5.00000 | 5.89000 |
| 60 | 5.00000 | 5.39000 |
| 61 | 450.00000 | 450.00000 |
| 62 | 450.00000 | 455.00000 |

Table 8 - Calibration data for calcium at 317.9 nm and 28 psi nebulizing pressure

LINEAR CURVE FITTING Y= .9734255298377394 X+-.5794226096644212
CORRELATION R= .9983987737576064

| Time (min) | Ca Standard (ppm) | Ca Observed (ppm) | Calculated Ca Conc. | S.D. for 95% Conf. Limit |
|---|---|---|---|---|
| II | X(II) | Y(II) | YC(II) | YS(II) |
| 1 | 100.00000 | 102.00000 | 96.76313 | 3.94756 |
| 2 | 90.00000 | 93.20000 | 87.02888 | 3.91881 |
| 3 | 70.00000 | 72.60000 | 67.56036 | 3.87694 |
| 4 | 50.00000 | 51.10000 | 48.09185 | 3.85643 |
| 5 | 40.00000 | 40.10000 | 38.35760 | 3.85431 |
| 6 | 30.00000 | 31.00000 | 28.62334 | 3.85763 |
| 7 | 20.00000 | 18.40000 | 18.88909 | 3.86637 |
| 8 | 10.00000 | 9.16000 | 9.15483 | 3.88051 |
| 9 | 1.00000 | 0.87000 | 0.39400 | 3.89779 |
| 10 | 0.00000 | 0.03200 | -0.57942 | 3.89997 |
| 11 | 100.00000 | 101.00000 | 96.76313 | 3.94756 |
| 12 | 90.00000 | 88.20000 | 87.02888 | 3.91881 |
| 13 | 70.00000 | 67.60000 | 67.56036 | 3.87694 |
| 14 | 50.00000 | 48.70000 | 48.09185 | 3.85643 |
| 15 | 40.00000 | 39.00000 | 38.35760 | 3.85431 |
| 16 | 30.00000 | 30.00000 | 28.62334 | 3.85763 |
| 17 | 20.00000 | 18.00000 | 18.88909 | 3.86637 |
| 18 | 10.00000 | 8.59000 | 9.15483 | 3.88051 |
| 19 | 1.00000 | 0.84400 | 0.39400 | 3.89779 |
| 20 | 0.00000 | 0.03800 | -0.57942 | 3.89997 |
| 21 | 100.00000 | 95.10000 | 96.76313 | 3.94756 |
| 22 | 90.00000 | 87.30000 | 87.02888 | 3.91881 |
| 23 | 70.00000 | 66.40000 | 67.56036 | 3.87694 |
| 24 | 50.00000 | 47.24000 | 48.09185 | 3.85643 |
| 25 | 40.00000 | 38.80000 | 38.35760 | 3.85431 |
| 26 | 30.00000 | 29.00000 | 28.62334 | 3.85763 |
| 27 | 20.00000 | 17.70000 | 18.88909 | 3.86637 |
| 28 | 10.00000 | 8.37000 | 9.15483 | 3.88051 |
| 29 | 1.00000 | 0.80600 | 0.39400 | 3.89779 |
| 30 | 0.00000 | 0.01200 | -0.57942 | 3.89997 |
| 31 | 100.00000 | 94.00000 | 96.76313 | 3.94756 |
| 32 | 90.00000 | 84.00000 | 87.02888 | 3.91881 |
| 33 | 70.00000 | 65.10000 | 67.56036 | 3.87694 |
| 34 | 50.00000 | 44.80000 | 48.09185 | 3.85643 |
| 35 | 40.00000 | 37.30000 | 38.35760 | 3.85431 |
| 36 | 30.00000 | 28.60000 | 28.62334 | 3.85763 |
| 37 | 20.00000 | 17.60000 | 18.88909 | 3.86637 |
| 38 | 10.00000 | 8.52000 | 9.15483 | 3.88051 |
| 39 | 1.00000 | 0.82500 | 0.39400 | 3.89779 |
| 40 | 0.00000 | -0.01200 | -0.57942 | 3.89997 |
| 41 | 100.00000 | 95.20000 | 96.76313 | 3.94756 |
| 42 | 90.00000 | 86.40000 | 87.02888 | 3.91881 |
| 43 | 70.00000 | 66.00000 | 67.56036 | 3.87694 |

Table 8 - Cont.

| Time (min) | Ca Standard (ppm) | Ca Observed (ppm) | Calculated Ca Conc. | S.D. for 95% Conf. Limit |
|---|---|---|---|---|
| 44 | 50.00000 | 47.60000 | 48.09185 | 3.85643 |
| 45 | 40.00000 | 38.00000 | 38.35760 | 3.85431 |
| 46 | 30.00000 | 30.20000 | 28.62334 | 3.85763 |
| 47 | 20.00000 | 18.30000 | 18.88909 | 3.86637 |
| 48 | 10.00000 | 8.44000 | 9.15483 | 3.88051 |
| 49 | 1.00000 | 0.81200 | 0.39400 | 3.89779 |
| 50 | 0.00000 | -0.01900 | -0.57942 | 3.89997 |
| 51 | 100.00000 | 94.40000 | 96.76313 | 3.94756 |
| 52 | 90.00000 | 85.30000 | 87.02888 | 3.91881 |
| 53 | 70.00000 | 64.60000 | 67.56036 | 3.87694 |
| 54 | 50.00000 | 46.60000 | 48.09185 | 3.85643 |
| 55 | 40.00000 | 37.70000 | 38.35760 | 3.85431 |
| 56 | 30.00000 | 27.90000 | 28.62334 | 3.85763 |
| 57 | 20.00000 | 17.50000 | 18.88909 | 3.86637 |
| 58 | 10.00000 | 8.10000 | 9.15483 | 3.88051 |
| 59 | 1.00000 | 0.79300 | 0.39400 | 3.89779 |
| 60 | 0.00000 | -0.01900 | -0.57942 | 3.89997 |

STANDARD DEVIATION OF THE SLOPE= 7.241893326147701E-3
STANDARD DEVIATION OF THE INTERCEPT= .3866180912757539
CALCULATED t-value OF THE SLOPE= 3.66954730834136
CALCULATED t-value OF THE INTERCEPT= 1.498695024209692
FOR 95% C.L. TABULATED VALUE = 2

Table 9 - Calibration data for iron at 372.0 nm and 28 psi nebulizing pressure.

LINEAR CURVE FITTING  Y= .977641202937593 X+ 4.933611345449382
CORRELATION R= .9995502369514758

| Time (min) | Fe Standard (ppm) | Ca Observed (ppm) | Calculated Ca Conc. | S.D. for 95% Conf. Limit |
|---|---|---|---|---|
| II | X(II) | Y(II) | YC(II) | YS(II) |
| 1 | 0.00000 | -0.09300 | 4.93361 | 23.32420 |
| 2 | 0.00000 | -0.09300 | 4.93361 | 23.32420 |
| 3 | 0.00000 | -0.09300 | 4.93361 | 23.32420 |
| 4 | 900.00000 | 900.00000 | 884.81069 | 23.32472 |
| 5 | 900.00000 | 872.00000 | 884.81069 | 23.32472 |
| 6 | 900.00000 | 838.00000 | 884.81069 | 23.32472 |
| 7 | 900.00000 | 885.00000 | 884.81069 | 23.32472 |
| 8 | 900.00000 | 864.00000 | 884.81069 | 23.32472 |
| 9 | 900.00000 | 893.00000 | 884.81069 | 23.32472 |
| 10 | 900.00000 | 876.00000 | 884.81069 | 23.32472 |
| 11 | 900.00000 | 860.00000 | 884.81069 | 23.32472 |
| 12 | 900.00000 | 878.00000 | 884.81069 | 23.32472 |
| 13 | 900.00000 | 899.00000 | 884.81069 | 23.32472 |
| 14 | 900.00000 | 902.00000 | 884.81069 | 23.32472 |
| 15 | 900.00000 | 895.00000 | 884.81069 | 23.32472 |
| 16 | 900.00000 | 897.00000 | 884.81069 | 23.32472 |
| 17 | 900.00000 | 888.00000 | 884.81069 | 23.32472 |
| 18 | 900.00000 | 890.00000 | 884.81069 | 23.32472 |
| 19 | 900.00000 | 886.00000 | 884.81069 | 23.32472 |
| 20 | 900.00000 | 902.00000 | 884.81069 | 23.32472 |
| 21 | 900.00000 | 881.00000 | 884.81069 | 23.32472 |
| 22 | 450.00000 | 459.00000 | 444.87215 | 23.07827 |
| 23 | 450.00000 | 469.00000 | 444.87215 | 23.07827 |
| 24 | 450.00000 | 460.00000 | 444.87215 | 23.07827 |
| 25 | 450.00000 | 455.00000 | 444.87215 | 23.07827 |
| 26 | 450.00000 | 463.00000 | 444.87215 | 23.07827 |
| 27 | 450.00000 | 450.00000 | 444.87215 | 23.07827 |
| 28 | 200.00000 | 203.00000 | 200.46185 | 23.15439 |
| 29 | 200.00000 | 203.00000 | 200.46185 | 23.15439 |
| 30 | 200.00000 | 198.00000 | 200.46185 | 23.15439 |
| 31 | 100.00000 | 102.00000 | 102.69773 | 23.22731 |
| 32 | 100.00000 | 103.00000 | 102.69773 | 23.22731 |
| 33 | 100.00000 | 101.00000 | 102.69773 | 23.22731 |
| 34 | 50.00000 | 53.70000 | 53.81567 | 23.27278 |
| 35 | 50.00000 | 52.80000 | 53.81567 | 23.27278 |
| 36 | 50.00000 | 54.90000 | 53.81567 | 23.27278 |
| 37 | 50.00000 | 52.90000 | 53.81567 | 23.27278 |
| 38 | 50.00000 | 52.70000 | 53.81567 | 23.27278 |
| 39 | 50.00000 | 54.20000 | 53.81567 | 23.27278 |
| 40 | 25.00000 | 25.50000 | 29.37464 | 23.29775 |
| 41 | 25.00000 | 25.70000 | 29.37464 | 23.29775 |

Cont. overleaf

Table 9 - Cont.

| Time (min) | Fe Standard (ppm) | Ca Observed (ppm) | Calculated Ca Conc. | S.D. for 95% Conf. Limit |
|---|---|---|---|---|
| 42 | 25.00000 | 26.20000 | 29.37464 | 23.29775 |
| 43 | 10.00000 | 10.80000 | 14.71002 | 23.31344 |
| 44 | 10.00000 | 11.20000 | 14.71002 | 23.31344 |
| 45 | 10.00000 | 11.20000 | 14.71002 | 23.31344 |
| 46 | 5.00000 | 5.21000 | 9.82182 | 23.31879 |
| 47 | 5.00000 | 5.11000 | 9.82182 | 23.31879 |
| 48 | 5.00000 | 5.39000 | 9.82182 | 23.31879 |
| 49 | 450.00000 | 439.00000 | 444.87215 | 23.07827 |
| 50 | 450.00000 | 454.00000 | 444.87215 | 23.07827 |
| 51 | 450.00000 | 453.00000 | 444.87215 | 23.07827 |
| 52 | 900.00000 | 886.00000 | 884.81069 | 23.32472 |
| 53 | 900.00000 | 865.00000 | 884.81069 | 23.32472 |
| 54 | 900.00000 | 874.00000 | 884.81069 | 23.32472 |
| 55 | 900.00000 | 890.00000 | 884.81069 | 23.32472 |
| 56 | 900.00000 | 869.00000 | 884.81069 | 23.32472 |
| 57 | 900.00000 | 882.00000 | 884.81069 | 23.32472 |
| 58 | 5.00000 | 5.77000 | 9.82182 | 23.31879 |
| 59 | 5.00000 | 5.89000 | 9.82182 | 23.31879 |
| 60 | 5.00000 | 5.39000 | 9.82182 | 23.31879 |
| 61 | 450.00000 | 450.00000 | 444.87215 | 23.07827 |
| 62 | 450.00000 | 455.00000 | 444.87215 | 23.07827 |
| 63 | 450.00000 | 459.00000 | 444.87215 | 23.07827 |

STANDARD DEVIATION OF THE SLOPE= 3.755502009453818E-3
STANDARD DEVIATION OF THE INTERCEPT= 2.221146285311467
CALCULATED t-value OF THE SLOPE= 5.953610730635372
CALCULATED t-value OF THE INTERCEPT= 2.221200547697177
FOR 95% C.L. TABULATED VALUE = 2

Table 10 - Calibration data for aluminum at 308.2 nm and 28 psi nebulizer pressure.

LINEAR CURVE FITTING  Y= 1.002671142857142 X+-1.029371428571428
CORRELATION R= .9994496491991492

| Time (min) | Al Standard (ppm) | Al Observed (ppm) | Calculated Al Conc. | S.D. for 95% Conf. Limit |
|---|---|---|---|---|
| II | X(II) | Y(II) | YC(II) | YS(II) |
| 1 | 0.00000 | 0.01200 | -1.02937 | 2.07560 |
| 2 | 100.00000 | 100.00000 | 99.23774 | 1.87745 |
| 3 | 100.00000 | 100.00000 | 99.23774 | 1.87745 |
| 4 | 100.00000 | 101.00000 | 99.23774 | 1.87745 |
| 5 | 100.00000 | 99.50000 | 99.23774 | 1.87745 |
| 6 | 100.00000 | 100.00000 | 99.23774 | 1.87745 |
| 7 | 100.00000 | 100.00000 | 99.23774 | 1.87745 |
| 8 | 100.00000 | 100.00000 | 99.23774 | 1.87745 |
| 9 | 50.00000 | 49.30000 | 49.10419 | 1.90977 |
| 10 | 50.00000 | 48.30000 | 49.10419 | 1.90977 |
| 11 | 50.00000 | 48.60000 | 49.10419 | 1.90977 |
| 12 | 0.00000 | -0.05400 | -1.02937 | 2.07560 |
| 13 | 100.00000 | 102.00000 | 99.23774 | 1.87745 |
| 14 | 100.00000 | 100.00000 | 99.23774 | 1.87745 |
| 15 | 100.00000 | 99.80000 | 99.23774 | 1.87745 |
| 16 | 100.00000 | 99.00000 | 99.23774 | 1.87745 |
| 17 | 100.00000 | 99.90000 | 99.23774 | 1.87745 |
| 18 | 100.00000 | 100.00000 | 99.23774 | 1.87745 |
| 19 | 100.00000 | 99.90000 | 99.23774 | 1.87745 |
| 20 | 100.00000 | 99.80000 | 99.23774 | 1.87745 |
| 21 | 50.00000 | 48.70000 | 49.10419 | 1.90977 |
| 22 | 50.00000 | 48.60000 | 49.10419 | 1.90977 |
| 23 | 50.00000 | 48.10000 | 49.10419 | 1.90977 |
| 24 | 50.00000 | 48.50000 | 49.10419 | 1.90977 |
| 25 | 50.00000 | 48.70000 | 49.10419 | 1.90977 |
| 26 | 100.00000 | 99.40000 | 99.23774 | 1.87745 |
| 27 | 100.00000 | 99.20000 | 99.23774 | 1.87745 |
| 28 | 100.00000 | 98.70000 | 99.23774 | 1.87745 |
| 29 | 100.00000 | 98.60000 | 99.23774 | 1.87745 |
| 30 | 100.00000 | 98.90000 | 99.23774 | 1.87745 |
| 31 | 100.00000 | 98.10000 | 99.23774 | 1.87745 |
| 32 | 100.00000 | 99.50000 | 99.23774 | 1.87745 |
| 33 | 100.00000 | 99.50000 | 99.23774 | 1.87745 |
| 34 | 100.00000 | 98.80000 | 99.23774 | 1.87745 |
| 35 | 100.00000 | 98.30000 | 99.23774 | 1.87745 |
| 36 | 100.00000 | 98.70000 | 99.23774 | 1.87745 |
| 37 | 100.00000 | 99.40000 | 99.23774 | 1.87745 |
| 38 | 100.00000 | 98.20000 | 99.23774 | 1.87745 |
| 39 | 100.00000 | 99.00000 | 99.23774 | 1.87745 |
| 40 | 100.00000 | 98.00000 | 99.23774 | 1.87745 |
| 41 | 100.00000 | 98.00000 | 99.23774 | 1.87745 |
| 42 | 100.00000 | 99.60000 | 99.23774 | 1.87745 |
| 43 | 100.00000 | 98.30000 | 99.23774 | 1.87745 |
| 44 | 100.00000 | 97.00000 | 99.23774 | 1.87745 |

STANDARD DEVIATION OF THE SLOPE= 5.135089156886563E-3
STANDARD DEVIATION OF THE INTERCEPT= .4644862908658687
CALCULATED t-value OF THE SLOPE= .5201745822777847
CALCULATED t-value OF THE INTERCEPT= 2.216150290792293
FOR 95% C.L. TABULATED VALUE = 2.021

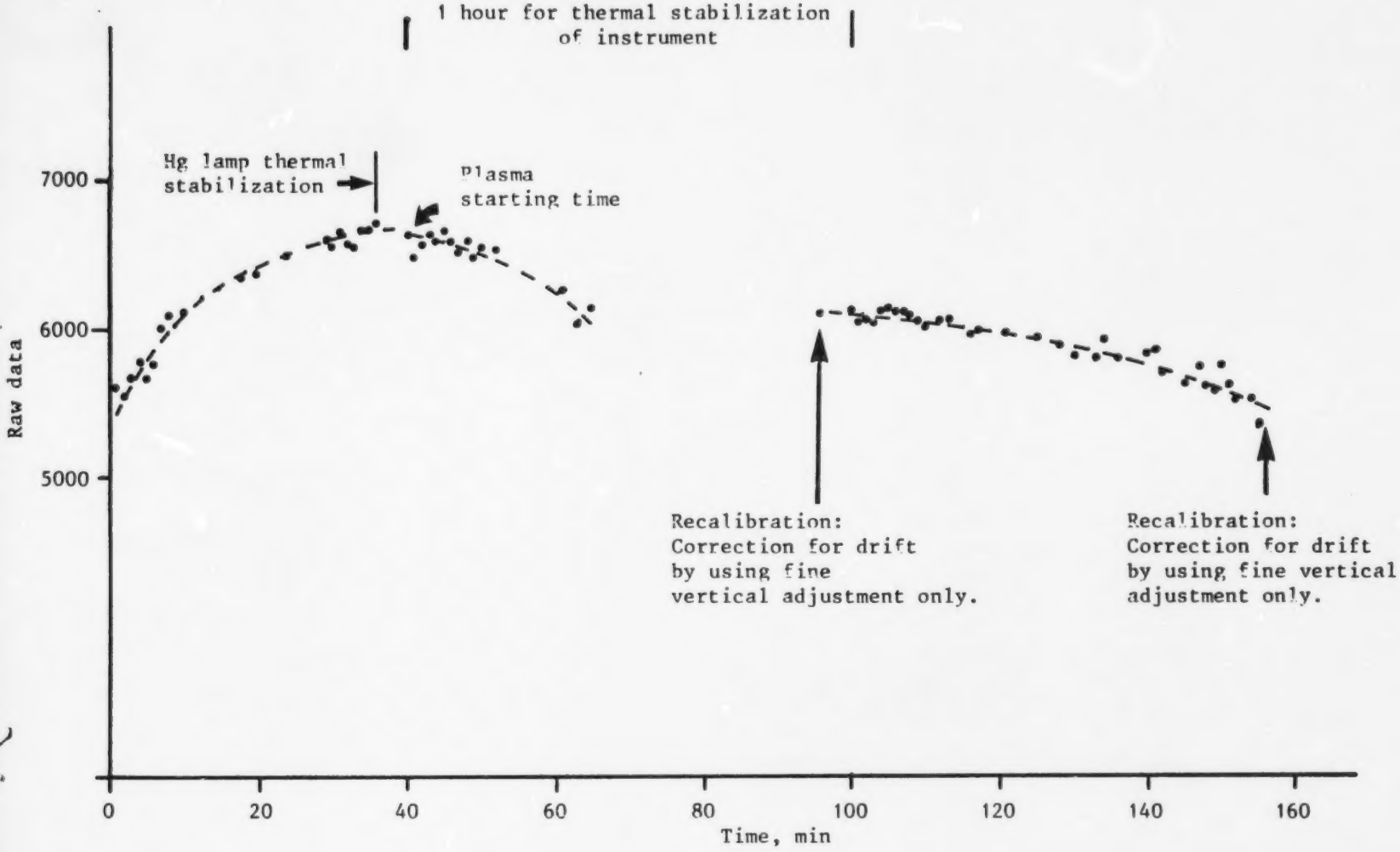


Fig. 1 - Optical and thermal stability test using a mercury lamp.

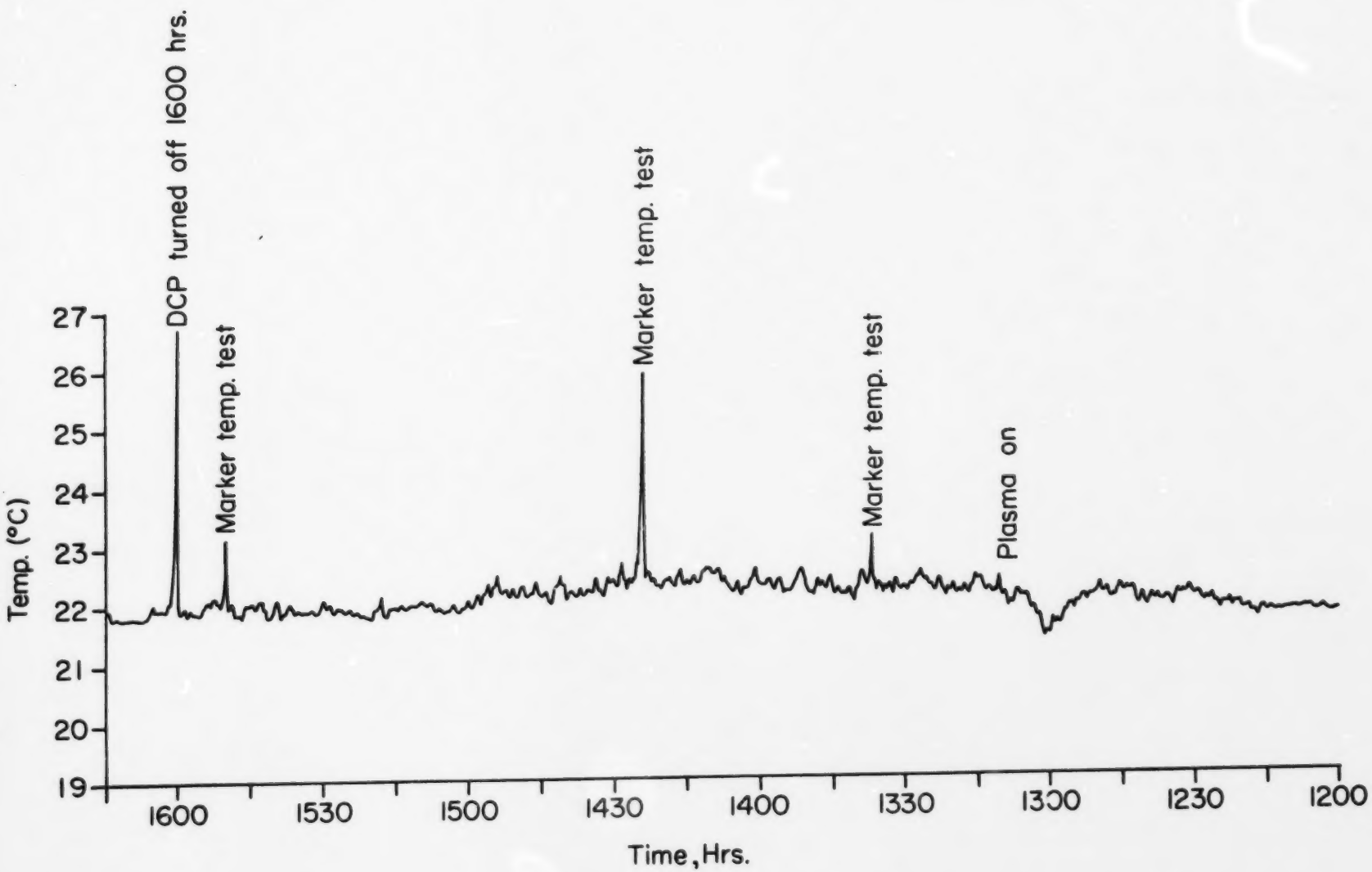


Fig. 2 - Room temperature fluctuations in the DCP room for a typical operating period.

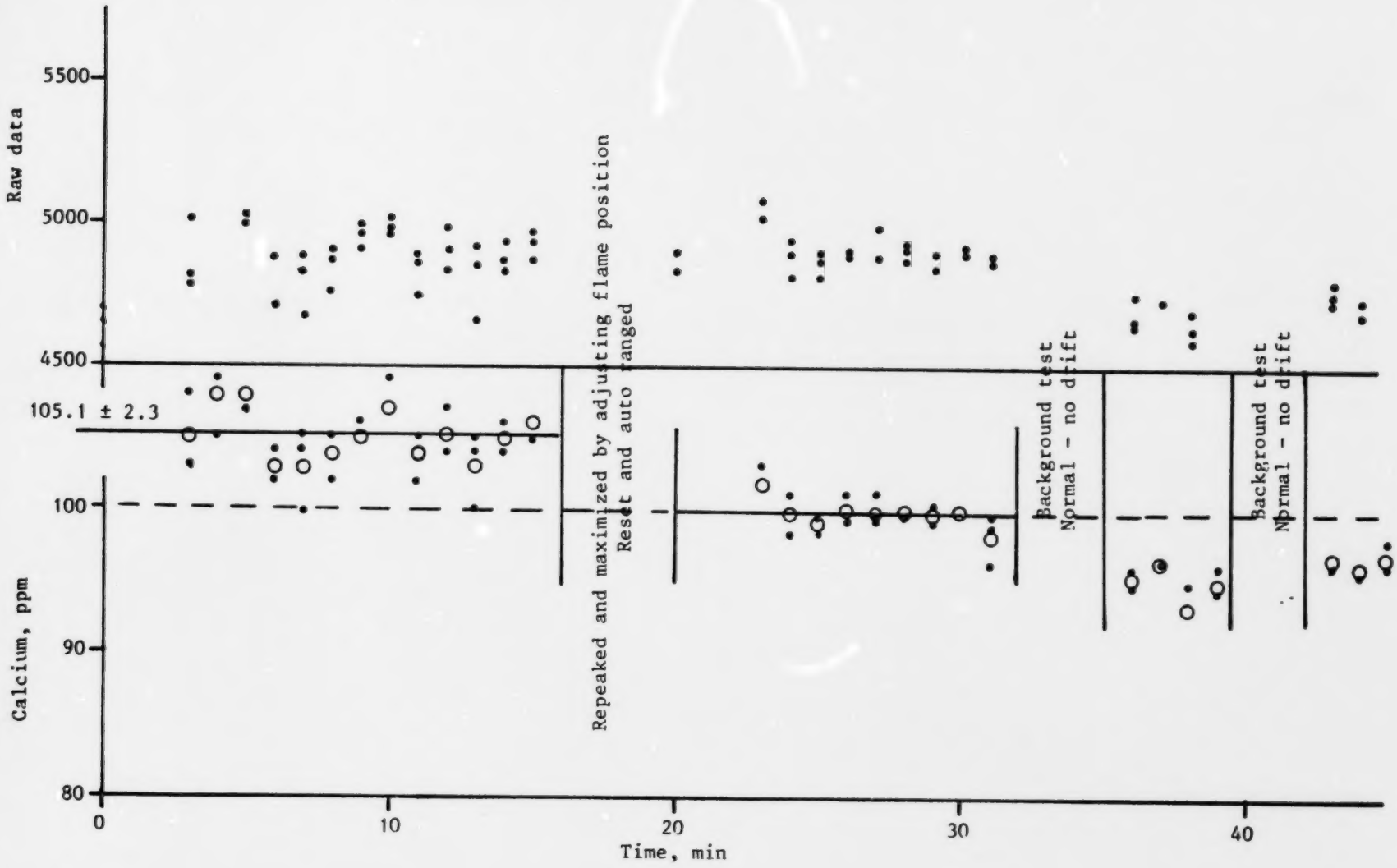


Fig. 3 - Stability test on calcium line at 317.9 nm observed at 100 ppm. (from Table 1.)

Cont. overleaf

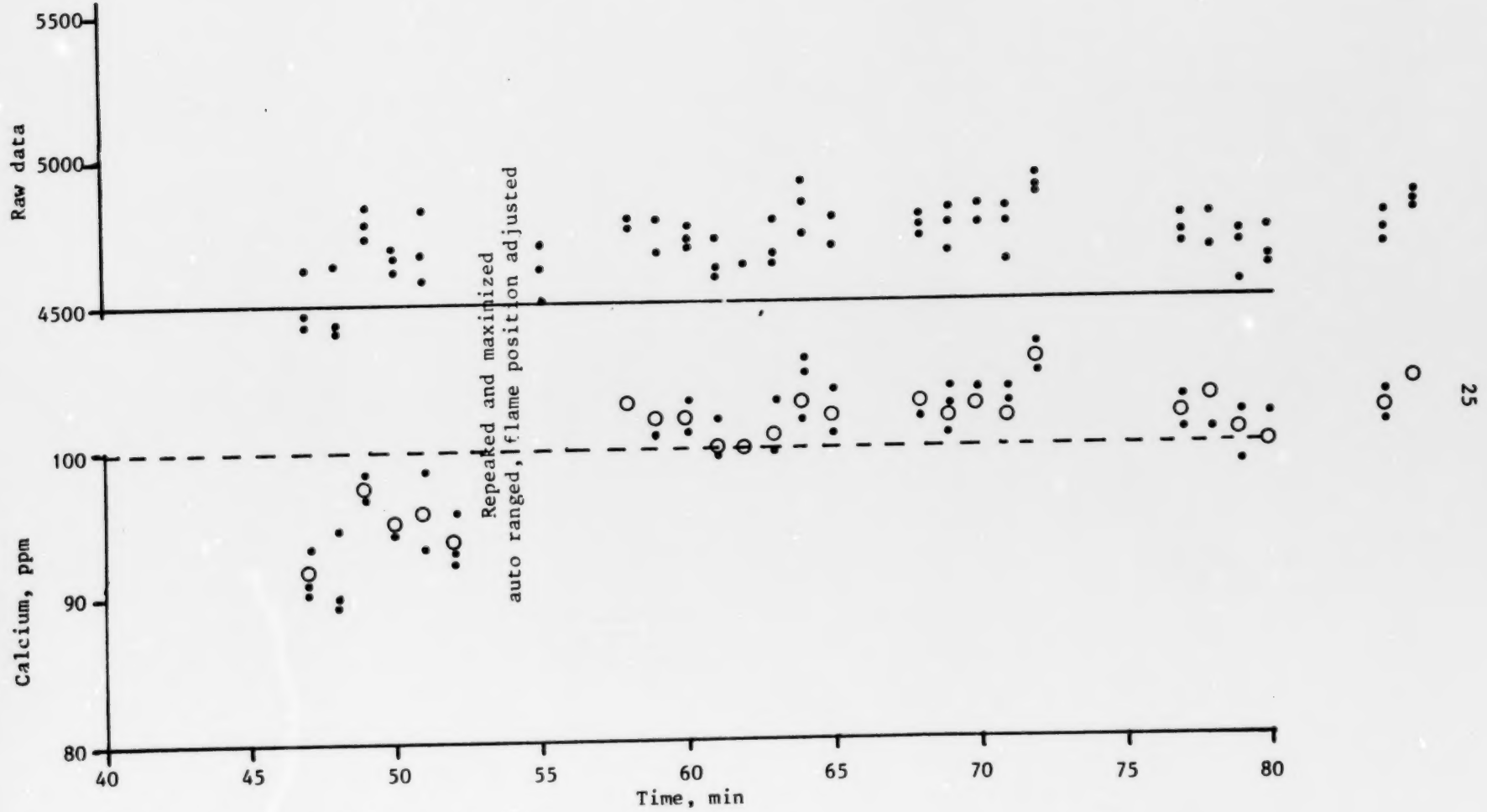
5500
5000
4500
100
90
80
Raw data
Calcium, ppm
Repeaked and maximized
auto ranged, flame position adjusted
40
45
50
55
60
65
70
75
80
Time, min
25

Fig. 3 - cont.

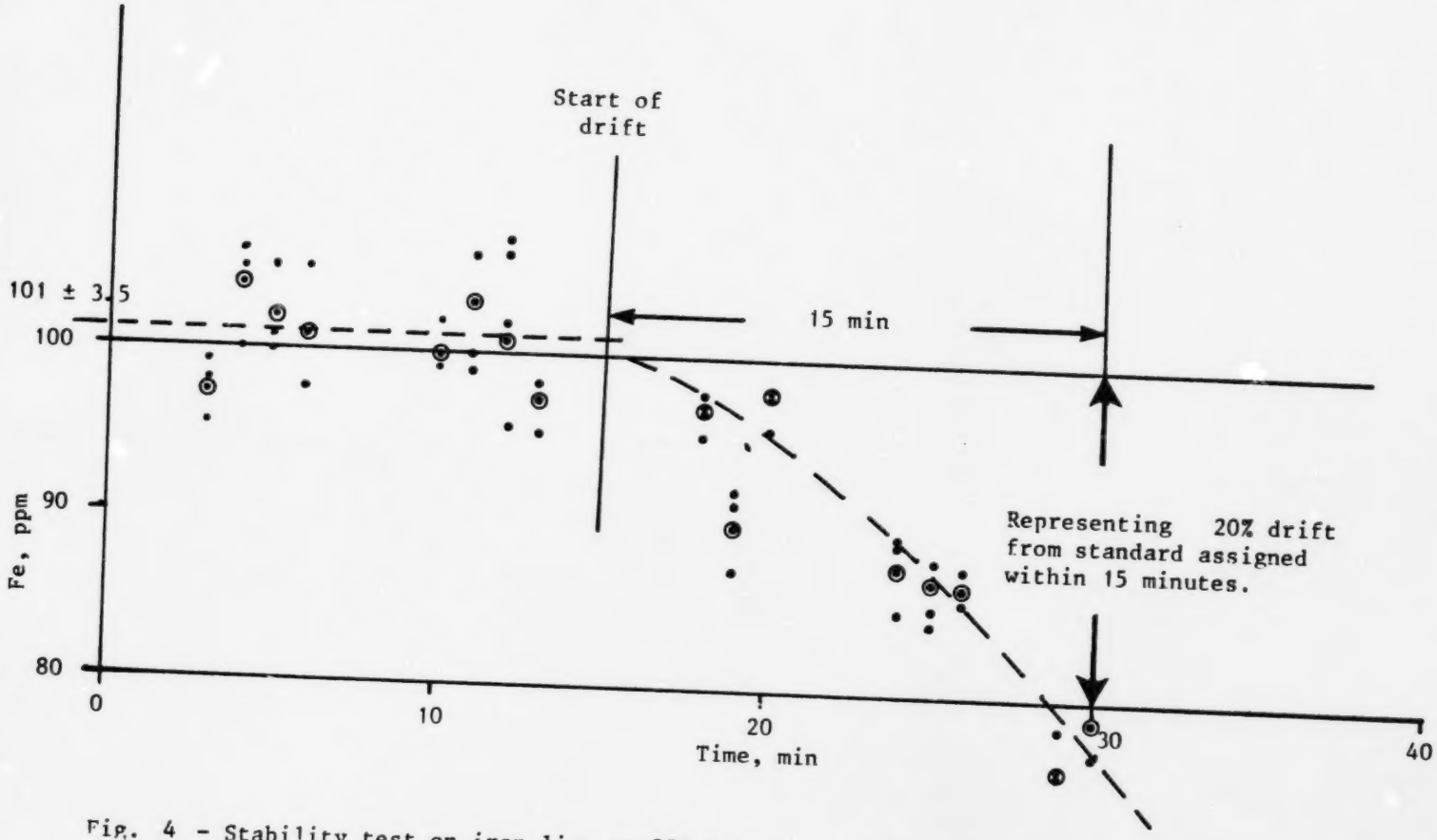


Fig. 4 - Stability test on iron line at 259.9 nm observed at 100 ppm concentration. (From Table 3.)

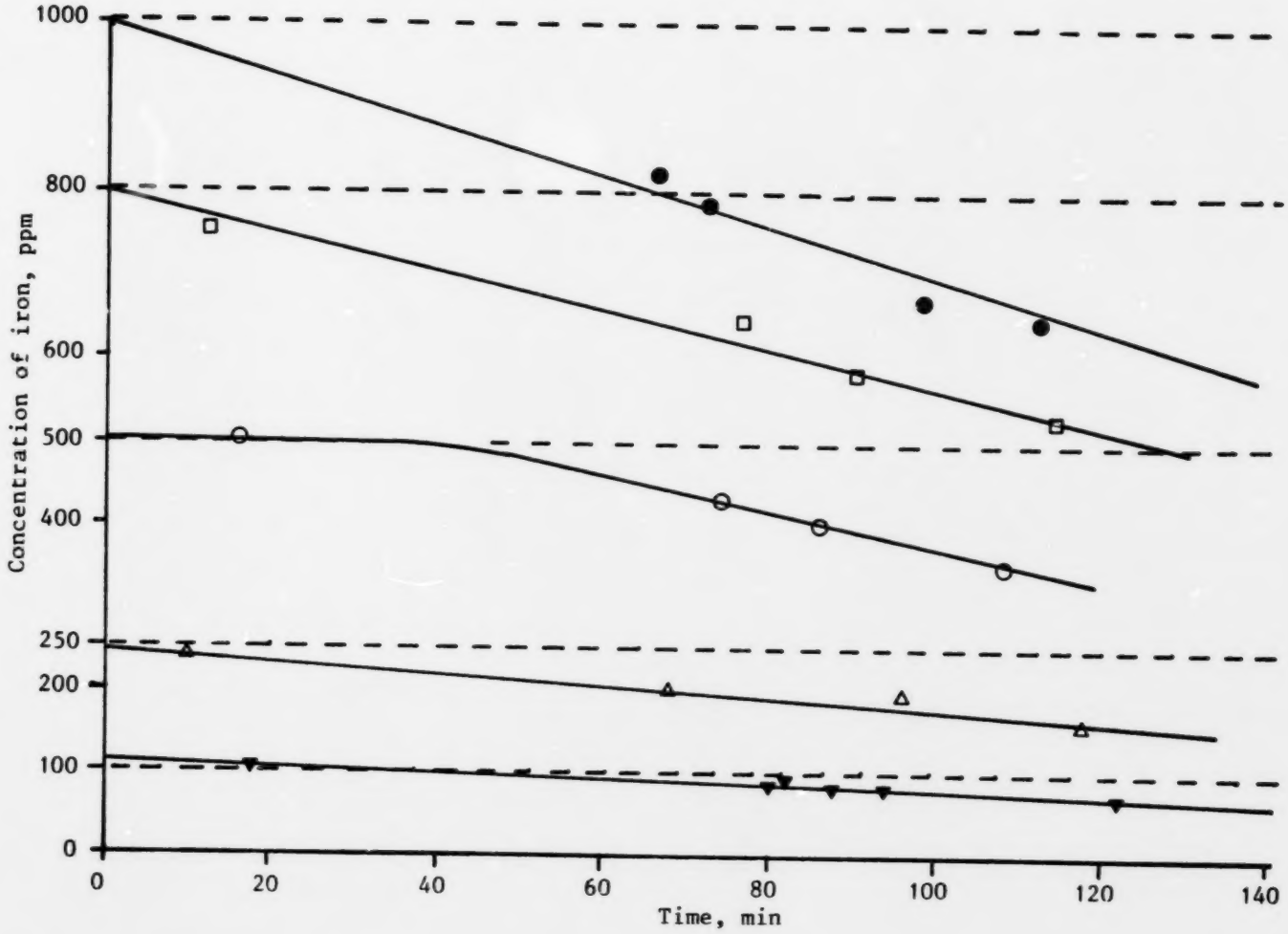


Fig. 5 - Stability test on iron line at 259.9 nm for various concentrations between 0-1000 ppm. Dotted line represents expected value, solid line represents observed value. (From Table 2.)

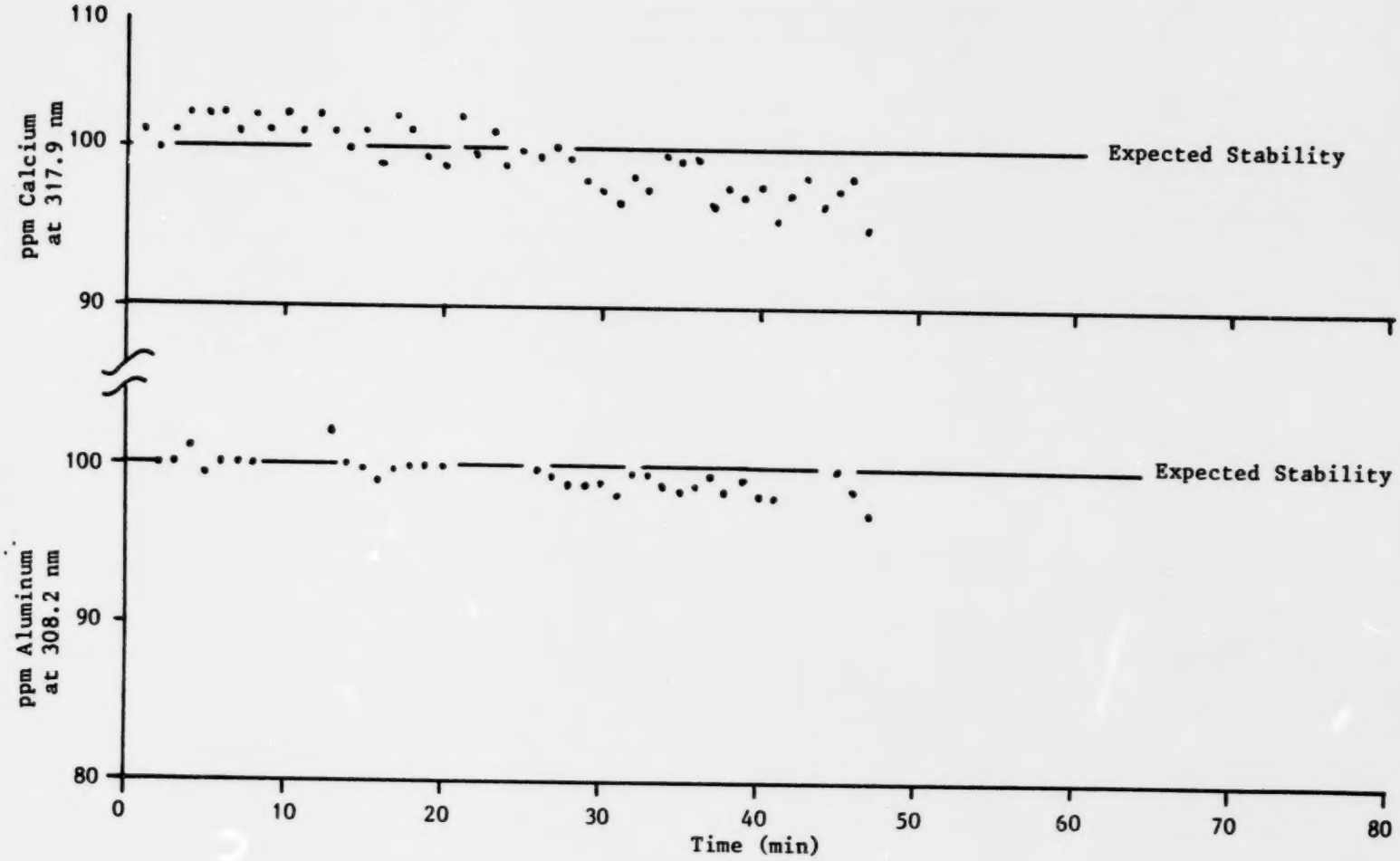


Fig. 6 - Aluminum and calcium stability test at 28 psi nebulizer pressure. (From Tables 5 and 6.)

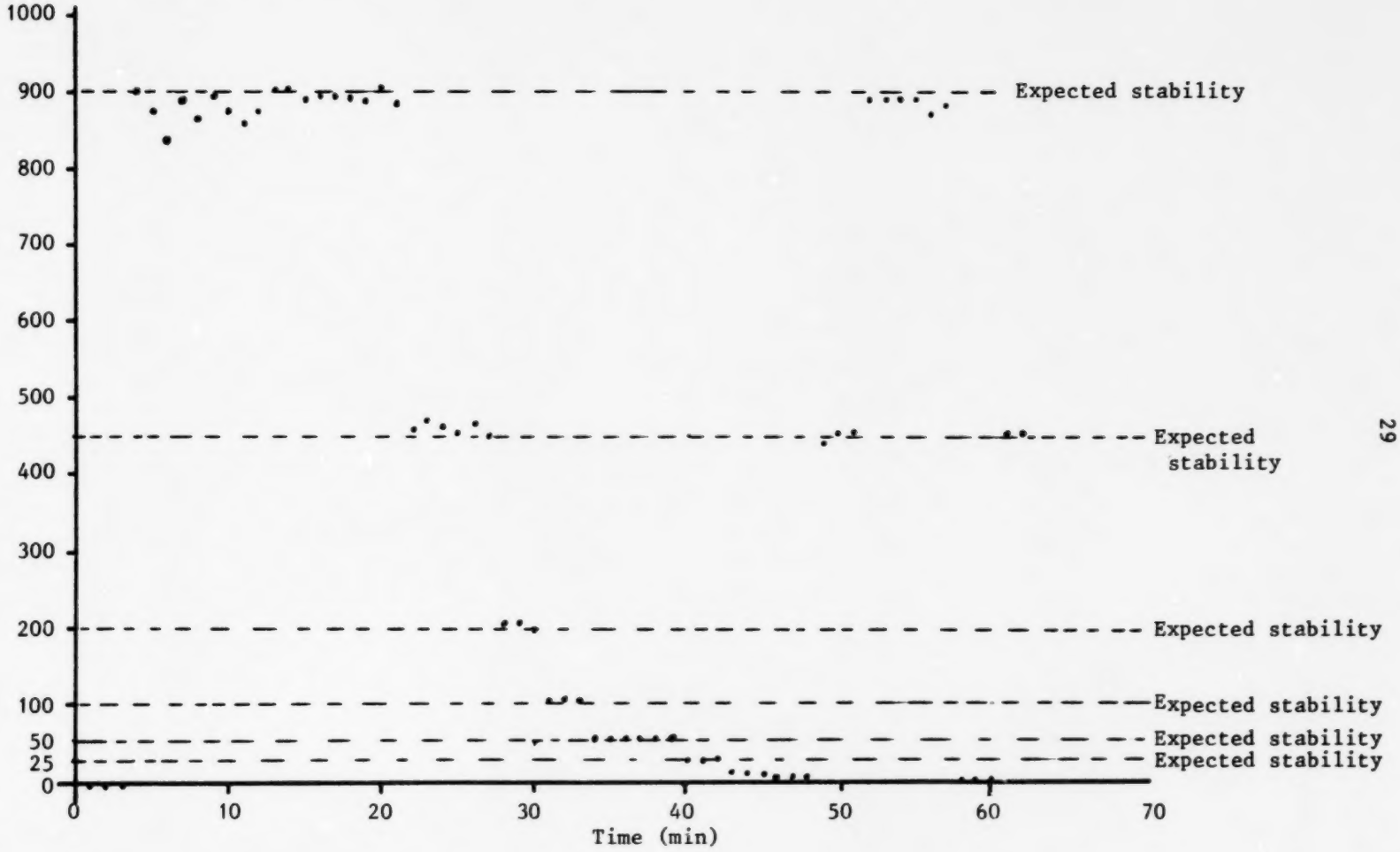


Fig. 7 - Iron stability test at 259.9 nm and 28 psi nebulizing pressure. (From Table 7.)

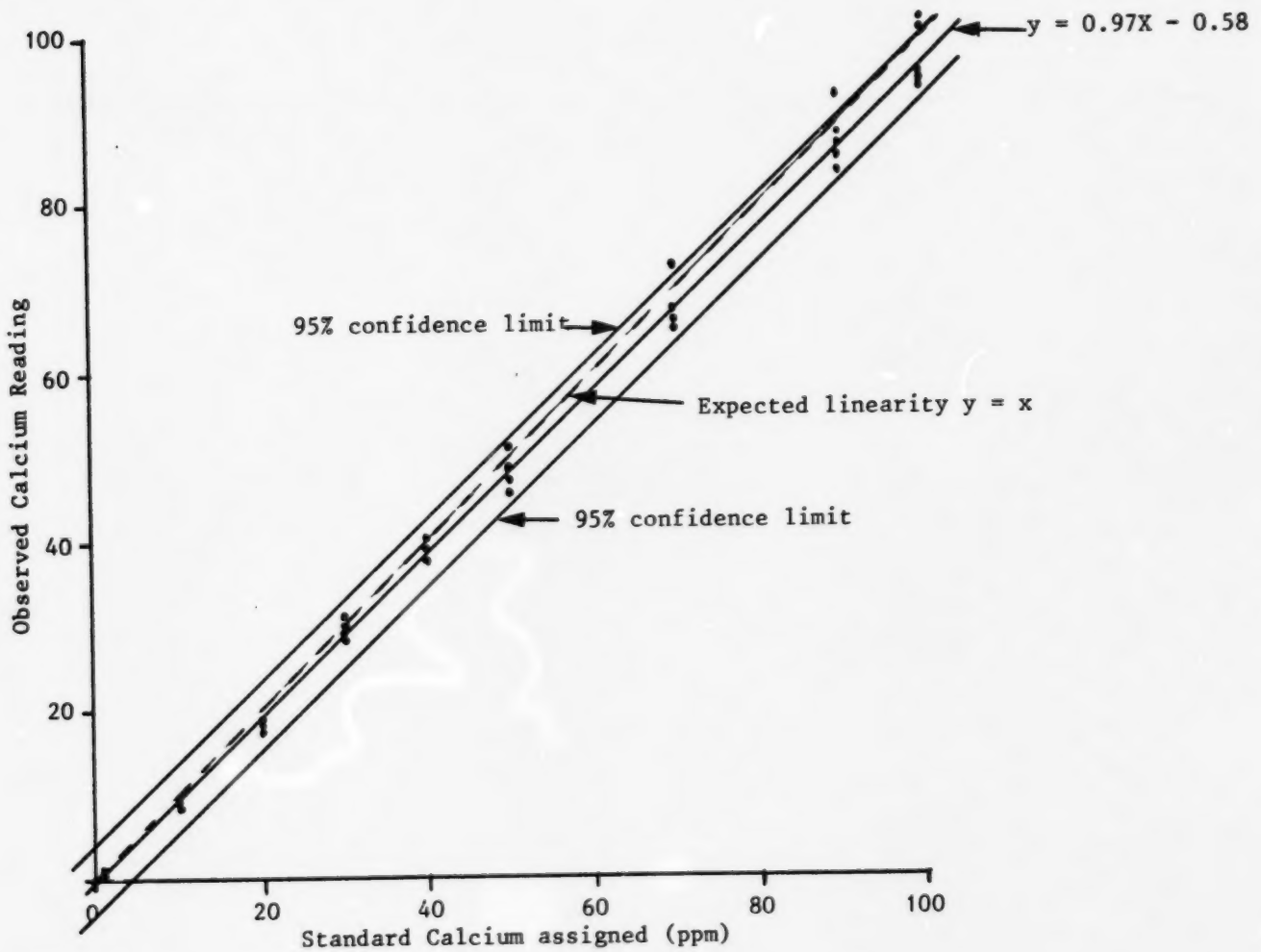


Fig. 8 - Calibration curve for calcium at 317.9 nm and 28 psi nebulizer pressure.

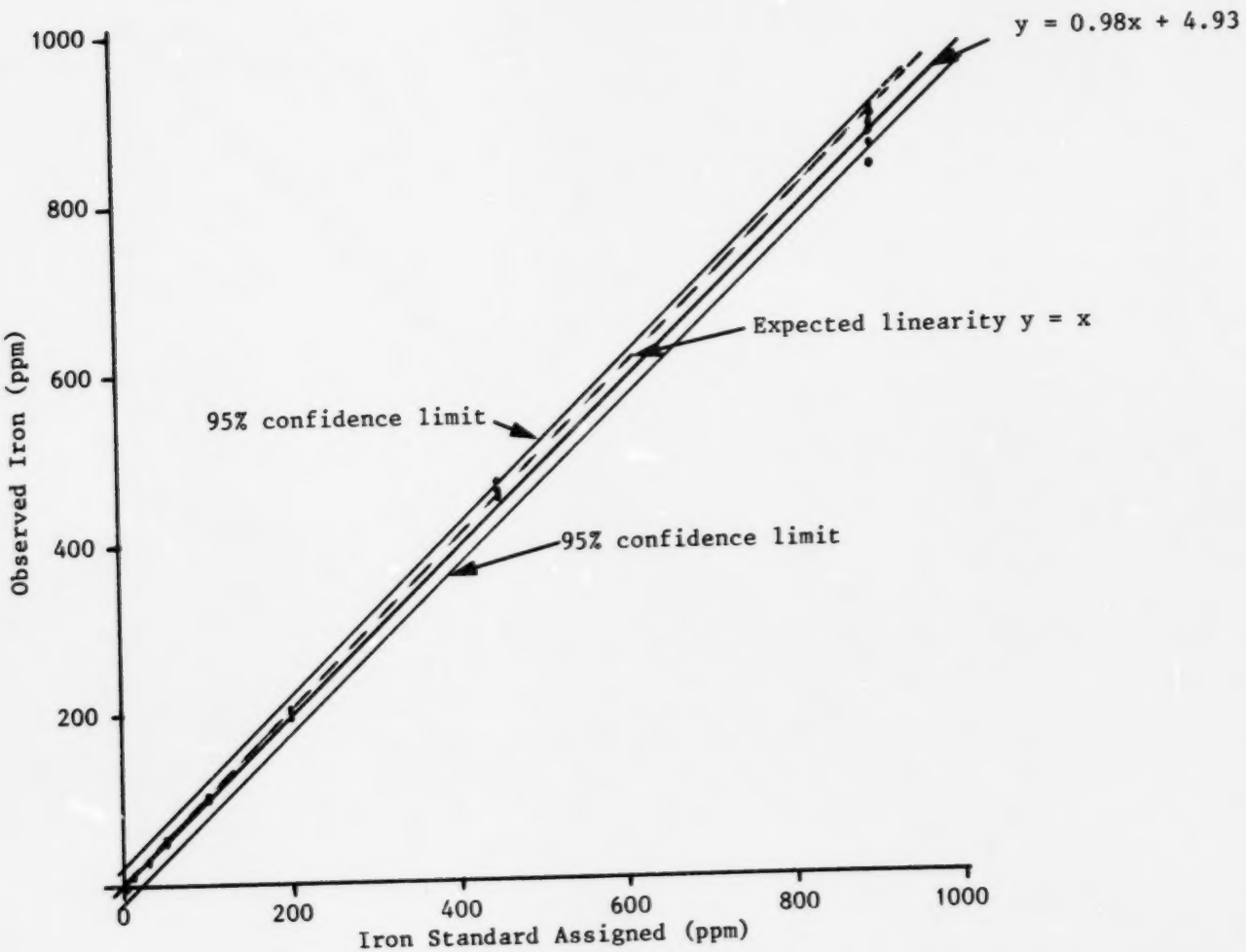


Fig. 9 - Iron calibration curve at 372.0 nm and 28 psi nebulizer pressure.

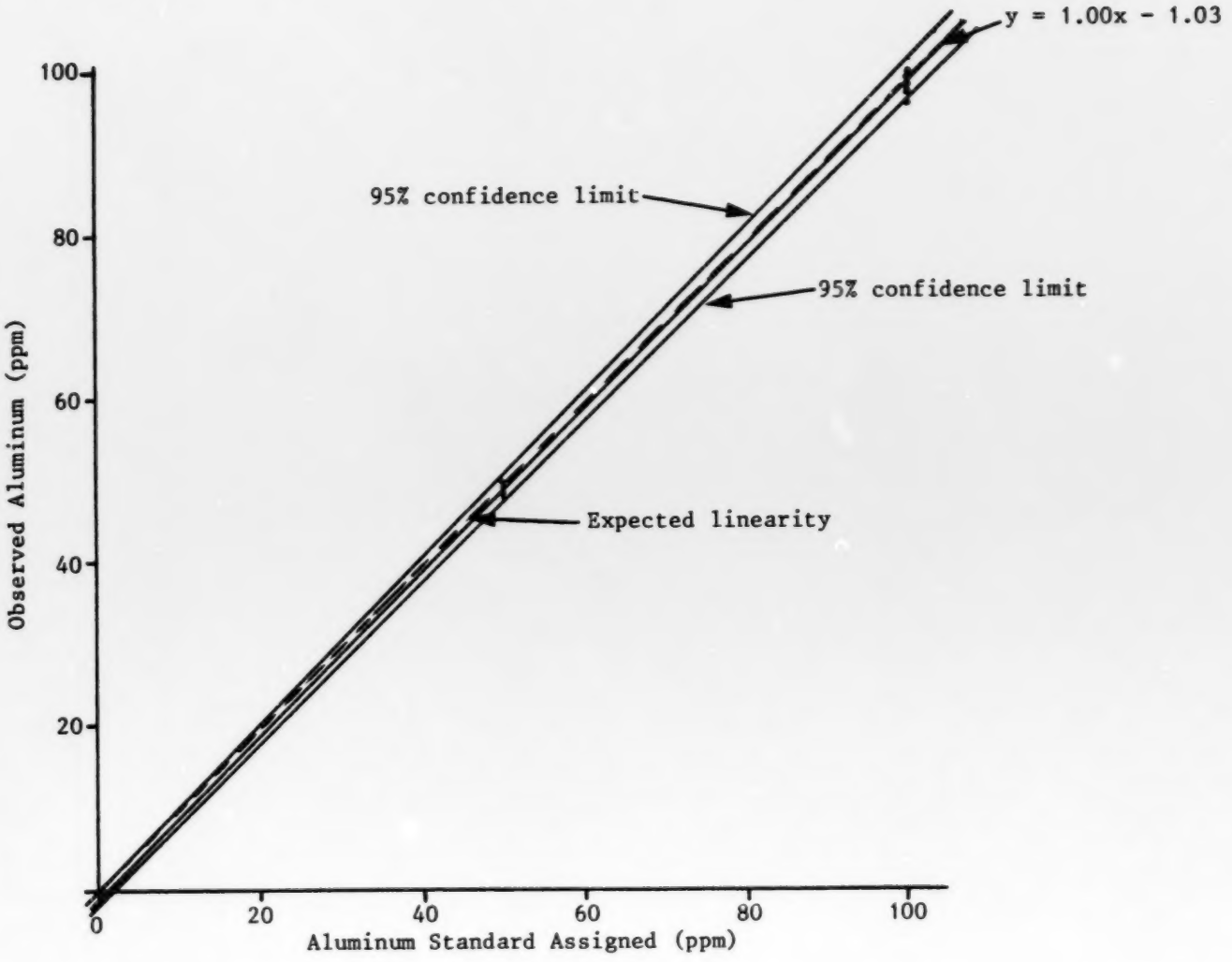


Fig. 10 - Aluminum calibration curve at 308.2 nm and 28 psi nebulizer pressure.